

## Novel synthesis and characterization of some new-2-(R) phenyl- 4-(- 4-bromo-2-fluoro benzylidene)-oxazol-5-ones

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### ABSTRACT

In the present study a series of some new 2-(substituted) phenyl- 4-(4-bromo-2-fluoro benz-ylidene)-oxazol-5-ones (2a-2j) were synthesized by the condensation of selected substituted benzoyl glycine (1a-1j) with 4-bromo-2-fluoro benzaldehyde in the presence of fused sodium acetate and acetic anhydride. The constitution of the newly synthesized compounds has been supported by their physical properties, elemental analysis, colour, m.p, IR spectral analysis data.

**Key words:** Substituted Oxazolones, Substituted Benzoyl Glycines, Substituted Benzaldehyde, Synthesis, Characterization, Spectral data.

### INTRODUCTION

The chemistry of oxazolone and its derivatives has been studied extensively for last few decades and has received important attraction in the synthesis of heterocycles, many of the substituted oxazolone and its derivatives possess a broad spectrum biological activity<sup>1</sup>.

Some of the novel substituted oxazolones have been reported to possess anti-microbial or anti-tumor<sup>2</sup>, anti-convulsant<sup>3</sup>, anti-bacterial<sup>4</sup>, analgesic<sup>5</sup> activities as well as bio-sensors coupling and photo sensitive composition devices of proteins<sup>6</sup>. Oxazol-5-ones are synthesized by the Erylenmeyer condensation as reported in the literature<sup>7</sup>.

Literature survey reveals that various oxazolones possess a broad spectrum of activities which are reflected by their use as anti-inflammatory<sup>8-9</sup>, anticancer<sup>10</sup>, antiobesity<sup>11</sup>, anti-depressant<sup>12</sup>.

In this paper the author reports here the synthesis of various substituted oxazol-5-ones has been synthesized from selected substituted benzoyl glycines with 4-bromo-2-fluoro benzaldehyde in the presence of fused sodium acetate as a support catalyst and acetic anhydride. Substituted benzoyl glycines, substituted oxazolones and their derivatives have been prepared by various workers in our laboratory<sup>13-18</sup>.

### EXPERIMENTAL

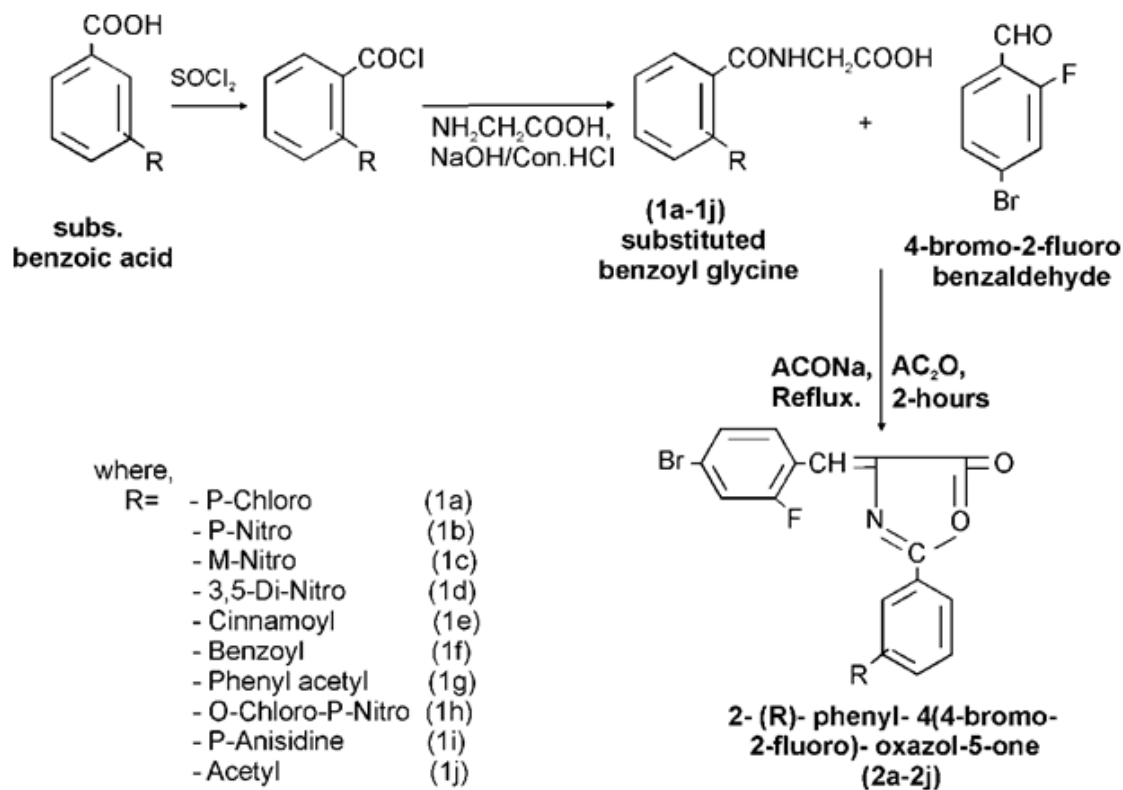
#### Material and Methods

#### All the recorded melting points were determined

In open capillary tubes and are uncorrected. All the chemicals and solvents used in the synthesis are of laboratory grade and solvents were purified. Completion of the reaction was monitored by TLC silica-gel-coated Al-Plates (Merck). IR(infrared) spectrum in KBr-disc method (cm<sup>-1</sup>) were recorded on Perkin-Elmer spectrum RX-1 FT-IR spectrophotometer at ST.John's College Agra. Elemental analysis, physical properties, colour, m.p, yield% of newly synthesized compounds are furnished in Table 1.

**Table 1: Physical and Analytical data of Newly Synthesized Compounds (2a-2j)**

codes	Molecular Formula	Molecular Weight	M.P °C	Yield %	Colour	% C cal. (found)	% H cal. (found)	%N cal. (found)
2a	C <sub>16</sub> H <sub>9</sub> N <sub>1</sub> O <sub>2</sub> Cl <sub>1</sub> Br <sub>1</sub> F <sub>1</sub>	380.62	188°	44.23	deep yellow	50.49 (50.50)	2.11 (2.13)	3.68 (3.72)
2b	C <sub>16</sub> H <sub>8</sub> N <sub>2</sub> O <sub>4</sub> F <sub>1</sub> Br <sub>1</sub>	391.17	164°	36.06	dark orange	49.12 (49.13)	2.06 (2.04)	7.16 (7.19)
2c	C <sub>16</sub> H <sub>8</sub> N <sub>2</sub> O <sub>4</sub> F <sub>1</sub> Br <sub>1</sub>	391.17	151°	29.97	deep orange	49.12 (49.10)	2.06 (2.05)	7.16 (7.12)
2d	C <sub>16</sub> H <sub>7</sub> N <sub>3</sub> O <sub>6</sub> F <sub>1</sub> Br <sub>1</sub>	436.17	232°	57.62	dirty yellow	44.06 (44.07)	1.61 (1.62)	9.63 (9.66)
2e	C <sub>16</sub> H <sub>11</sub> N <sub>1</sub> O <sub>2</sub> F <sub>1</sub> Br <sub>1</sub>	372.21	195°	63.72	red	58.08 (58.06)	2.97 (2.99)	3.76 (3.72)
2f	C <sub>16</sub> H <sub>9</sub> N <sub>1</sub> O <sub>2</sub> F <sub>1</sub> Br <sub>1</sub>	346.17	138°	60.73	sunrise orange	55.51 (55.49)	2.62 (2.64)	4.04 (4.01)
2g	C <sub>17</sub> H <sub>11</sub> N <sub>1</sub> O <sub>2</sub> F <sub>1</sub> Br <sub>1</sub>	360.20	158°	48.98	dark orange	56.68 (56.66)	3.07 (3.04)	3.88 (3.91)
2h	C <sub>16</sub> H <sub>7</sub> N <sub>2</sub> O <sub>4</sub> Cl <sub>1</sub> F <sub>1</sub> Br <sub>1</sub>	425.62	211°	55.74	deep mehron red	45.15 (45.17)	1.65 (1.63)	6.58 (6.61)
2i	C <sub>16</sub> H <sub>10</sub> N <sub>2</sub> O <sub>2</sub> F <sub>1</sub> Br <sub>1</sub>	361.18	250°	59.79	dark fire red	53.20 (50.22)	2.79 (2.77)	7.75 (7.71)
2j	C <sub>11</sub> H <sub>7</sub> N <sub>1</sub> O <sub>2</sub> F <sub>1</sub> Br <sub>1</sub>	284.10	197°	39.68	dark red	46.50 (46.51)	2.48 (2.46)	4.93 (4.89)



**General procedure for the synthesis of substituted benzoyl glycine (1a-1j)**

A mixture of substituted benzoic acid (0.025 mole) and thionyl chloride (0.05 mole) in r.b flask having calcium chloride guard tube to the top of the condenser, contents were refluxed on boiling water-bath for about 3-hours, after cooling the liquid was distilled to remove the excess of  $\text{SOCl}_2$ , glycine (0.025 mole) dissolved in 2N NaOH (25 ml) and substituted acid chloride (0.025 mole) was added in small portions and the flask was shaken vigorously after each addition. The obtained clear solution was acidified with concentrated HCl, thus the obtained solid was collected, filtered, recrystallized from hot water.

**General procedure for the synthesis of 2-(substituted) phenyl- 4-(4-bromo-2-fluoro benzylidene)-oxazol-5-one (2a-2j)**

A mixture of substituted benzaldehyde (4-bromo-2-fluoro; 0.0025 mole) and substituted benzoyl glycine (1a-1j; 0.0025 mole), (0.20 gm) of anhydrous sodium acetate and (0.7 ml) of acetic anhydride was heated on a water-bath for 2-hours, the contents were cooled and then treated with ice-cold water, filtered, again washed with distilled water. The obtained product was purified by recrystallization from absolute ethanol and ethyl acetate.

**Table 2: Characterization IR( $\nu$  in  $\text{cm}^{-1}$ ) data of newly synthesized compounds**

codes	Furan-CH $\text{cm}^{-1}$ stretching	Ar.-CH $\text{cm}^{-1}$ stretching	-C=O $\text{cm}^{-1}$ stretching	-N=C $\text{cm}^{-1}$ stretching	-C=C $\text{cm}^{-1}$ stretching	C-O-C $\text{cm}^{-1}$ stretching	CH=C $\text{cm}^{-1}$ stretching	C-Cl $\text{cm}^{-1}$ linkage
2a	3235.0	3052.2	1685.9	1639.2	1592.2	1176.5	973.0	682.3
2f	3190.0	3068.5	1722.6	1639.2	1596.8	1171.6	988.7	694.0

**RESULTS AND DISCUSSION**

The infrared spectra of newly synthesized compounds have been recorded in the frequency region 4000-500  $\text{cm}^{-1}$ . The IR (in Kbr-disc method)

**Spectral data are recorded in the Table-2**

The IR spectrum of compounds (2a,2f) showed absorption bands at 3235.0 and 3190.0  $\text{cm}^{-1}$  indicating Furan ring -CH, absorption at range 3052.2 and 3068.5  $\text{cm}^{-1}$  shows Ar. CH str., while absorption bands at 1685.9 and 1722.6  $\text{cm}^{-1}$  reveals -C=O stretching vibrations, stretching vibrations at

1639.2 and 1639.2  $\text{cm}^{-1}$  confirm the presence of (oxazolone ring -N=C), while the stretching vibrations at 1592.2 and 1596.8  $\text{cm}^{-1}$  indicates C=C, absorption spectrum at 1176.5 and 1171.6  $\text{cm}^{-1}$  shows C-O-C str., absorption bands at 973.0 and 988.7  $\text{cm}^{-1}$  indicates the presence of CH=C str., while absorption at 682.3 and 694.0  $\text{cm}^{-1}$  indicates the presence of C-Cl linkage. The above observations are sufficient to support the assigned structure of the newly synthesized compounds (2a,2f) and other compounds (2b-2e,2g-2j). The oxazolones are coloured solids and having high melting points.

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